

Effect of nano-SiO₂ particles on properties of cement mortar applicable for ferrocement elements

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Abstract

In this study the mechanical properties (by compressive and flexural strength tests), durability (by water absorption test), and microstructural properties of interfacial transition zone (ITZ) (by Scanning Electron Microscopy and Atomic Force Microscopy tests) of mortars applicable for the casting of ferrocement elements reinforced with nano-SiO₂ particles are investigated. The parameters of this study include the low replacement ratio of nano-SiO₂ particles respect to cement in Ordinary Portland Cement (OPC) mortar mixture (including 1%, 2% and 3%), water to binder ratio (including 0.35, 0.4 and 0.5), and also sand to binder ratio (including 2 and 2.5).

The results have shown that the cement mortars containing nano-particles have reasonably higher strength, low water absorption and denser ITZ compared to those of the OPC ferrocement mortars. Furthermore, along with increasing the W/CM, the performance of silica nano-particles has been reduced. Besides, using higher S/CM was followed by strength loss in both categories of mixtures including with and without silica nano-particles. However, distinctive strengthening trend was not observed in mixtures with different S/CM (by holding the other variables constant such as silica nano-particles).

Keywords: Ferrocement mortar; nano-SiO₂ particles; mechanical properties; water absorption; ITZ.

1. Introduction

Ferrocement is a type of thin wall reinforced concrete commonly constructed of hydraulic cement mortar reinforced with closely spaced layers of continuous and relatively small size wire mesh. The most common type of reinforcement is steel mesh. Other suitable materials such as selected organic, natural, or synthetic fibers may be combined with metallic mesh [1-3]. Compared to reinforced concrete, ferrocement has homogeneous-orthotropic properties in two directions. It exhibits high tensile strength, high modulus of rupture and superior cracking performance.

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In addition, because of the high specific surfaces of ferrocement meshes, larger bond forces develop with the matrix resulting in smaller crack spacing and width [3]. Applications of ferrocement are numerous, especially in structures or structural components where self-help or low levels of skills are required [1].

There has been increasing activity with ferrocement construction throughout the world [4-7]. For example, It is the promising composite material for the prefabrication and the industrialization of the building industry [8] and proved as excellent material for low cost housing [9, 10], and as encasement of lightweight core materials to produce high performance lightweight structural sandwich panels [11]. Moreover, besides boats and marine structures, ferrocement is used for water tanks, grain silos, flat or corrugated roofing sheets, irrigation channels, and the like [1].

In the present century which is replete with numerous innovative scientific advancements, nanotechnology is introduced as one of the most important scientific and technologic achievements of humankind. Besides, due to interdisciplinary nature of nanotechnology, it is a matter of extreme importance.

The nature of this technology is in a way which has made it applicable in all the fields of science and technology. Along with the involvement of nano science and technology in materials science and chemistry, nano-materials were offered to the market as basic products of nanotechnology.

Because nano-particles have shown singular behavior as principal elements of nano-materials, the application of nano-particles in different industries is increasing with a significant speed. Accordingly, the concrete industry was not an exception. It has been observing the entrance of different nano-particles in the matrix of cement-based materials. Based on previous researches, silica nano-particles have been introduced as the most compatible type of nano-particles applicable in concrete industry [12-14].

Various performances of silica nano-particles in the matrix of cement-based materials, high marketing accessibility, and their more suitable price compared to other types of nano-particles are some of the determining factors of their superiority over other nano-particles.

In addition, the use of nano-particles is a way to further development of the particle packing concept and manipulation of particle size distribution [15]. If the water content is kept constant, as in the actual conditions, an increase of nano-silica content will promote the packing of particles, decreasing the volume between them and decreasing the free water [16].

Furthermore, silica nano-particles can enhance the microstructural properties of cement-based materials due to their super pozzolanic behavior. Micro and nano filling effect, quick pozzolanic reaction with protlandite, and consequently the formation of dense Calcuim-Silicate-Hydrate, limiting the growth of portlandite crystals (CH), and promoting the hydration reaction are some of the influences of silica nano-particles [17].

Therefore, we will observe increase in strength and durability of cement-based materials if we add sufficient amount of these particles [18-21].

The considerable point in this study is application of low dosages of nano-particles as replacement of cement. Regarding our incapability in achieving a homogenous distribution of these particles and also risk of agglomeration, high dosage application of these materials would not be effective in the matrix of cement-based materials. Besides, regarding the cost of these materials, application of dosages lower than optimum content will be economically helpful.

Because the matrix in ferrocement has 95% or more pronounced influence on the behaviour of final product [22], which entirely depends upon the composition of the mortar mix, thus the properties of mortar mix like compressive strength, flexural strength and water absorption are very important to consider during the design of thin ferrocement structural elements.

Therefore, this experimental study is aimed to investigate the compressive strength, flexural strength, strength development, water absorption and microstructural properties of ferrocement mortars by incorporating different dosages of silica nano-particles as partial replacement of cement. Also, superplasticizer was applied as water reducing agent to provide similar flowability (150-180 mm according to Mini Slump test [23]) and pumpability.

2. Experimental program

2.1. Materials

Locally available ordinary Portland cement (Type I-425) complying with the Iranian national standards 389 was used. The colloidal nano-SiO₂ (with solid content of 30%) was used in this study. These chemical compositions and physical properties are summarized in Table 1.

Application of colloidal nano-particles aids better dispersion of nano-particles in the concrete matrix and decreases agglomeration of nano-particles which improves nano-particles performance in concrete [16].

Washed natural river sand with a fineness modulus of 3.2, a specific gravity of 2.74 g/cm³, and water absorption of 1% was adopted for fine aggregate. The grading of fine aggregate is considered in accordance with the requirements of ACI 549R-97 [1]. The water reducer superplasticizer (naphthalene-type with a solid content of 40%) is employed to aid the dispersion of nano-particles in mortar matrix and achieve good workability of mortar (because addition of nano-SiO₂ particles causes more viscose mortar than mortar without nano-SiO₂ particles) [17, 18].

TABLE 1: CHEMICAL AND PHYSICAL CHARACTERISTICS OF BINDERS

Items	Chemical composition (%)	
	OPC	Nano-SiO ₂
SiO ₂	21.4	99.9
Al ₂ O ₃	6	-
Fe ₂ O ₃	3.4	-
CaO	64	-
MgO	1.8	-
SO ₃	1.4	-
K ₂ O+Na ₂ O	1	-
LOI	3	2.8
Physical properties		
Specific gravity (g/cm ³)	3.15	1.2
Specific surface (m ² /g)	0.37	100±20
Average particle size	15 µm	35±5 nm

2.2. Experimental variables, procedure and specimen preparation

Single Two series of mixtures with different sand to cementitious materials (S/CM) ratio (2 and 2.5) are considered.

Each series has three water to cementitious materials (W/CM) ratios (0.35, 0.4 and 0.5).

The binder content (cementitious materials) is taken as the sum of cement and nano-silica particles. For each specific S/CM and W/CM ratio, four different dosages of nano-particles are used as replacement of cement (0%, 1%, 2% and 3% to the weight of cement). So, 24 mixes, as listed in Table 2, are studied.

To fabricate ferrocement mortars with and without nano-particles, the mixtures were mixed in a rotary mixer. Nano-particles are not easy to disperse uniformly due to their high surface energy.

Accordingly, based on trials and errors and by taking advantage of previous experiments [17], mixing was performed as follows:

1. The colloidal nano-SiO₂ particles (if applicable) and superplasticizer were stirred with the mixing water.
2. The cement were added to the mixer and mixed at low speed for 30 s.
3. Mixing at low speed, the moist sand (sand in saturated surface dry mood) was added gradually to the mixture during another 30s.
4. The mixture was allowed to rest for 90 s and then mixed for 2 min at medium speed to achieve good workability.

To investigate the workability of mixtures and effects of nano-particles in mortars, Mini Slump Flow test was conducted [23]. We tried our best to provide a similar situation for all the mixes in case of rheological properties (150-180 mm).

By changing the volume of Superplasticizer, the flowability of mixes varies. If the mixes approximately achieve the required diameters, then, they are ready to be poured in the moulds for curing and finally mechanical tests. The different volumes of Superplasticizer are also expressed in table 2.

After mixing, the well-mixed mortar was poured into oiled molds to form cubes of size 50 mm for compressive strength at different ages (at 7 and 28) and water absorption testing (at 28 days), and prisms of size 40×40×160 mm for flexural strength testing (at 28 days). The samples were demolded after 24 h and then cured in water tank (temperature 20±2°C) for 7 and 28 days.

Compressive and flexural strength at different ages of 7 and 28 days were determined according to ASTM C109-02 and ASTM C 348-02, respectively. Also, after the mechanical tests at 28 days, the crushed specimens were selected and prepared for Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM) tests to observe the effect of nano-particles on properties of ITZ.

Moreover, water absorption test was conducted only for mixtures with W/CM=0.4 according to BS 1881: Part 122-1983. In this test, 28-days specimens were put in oven at 105 °C for 72 hours. After a gradual cooling period in ambient environment (22± 0.5 °C), specimens were immersed in water for 30 minutes.

The value of absorption will be calculated according to equation 1 by measuring the dry weight (before immersing in water) and wet weight (after 30 min immerse). The specimen should be dried with towel before weighting the wet weight to procure a similar SSD condition.

$$WA = \frac{W_w - W_d}{W_d} \times 100 \quad (\text{Eq. 1})$$

In above equation, W_A , W_w , and W_d are water absorption percentage, wet weight, and dry weight, respectively.

Finally, by visual examination of fractured specimens under the compressive strength test and attributing each fractured specimen to different stability classification, the following comparative criterion was considered.

After conducting the compressive strength, specimen with lowest distortion was assigned to point 5, and point 1 was given to the most distorted specimen. Other specimens were relatively marked between 1 and 5.

Accordingly, dimensional stability of specimens was investigated in both 7 and 28 days.

TABLE 2: MIX PROPORTION OF THE SPECIMENS

Mix No.	S/CM	W/CM	Cement (g)	Nano-SiO ₂ (g)	Superplasticizer/CM	Sand (g)
1	2	0.35	450	0	0.10	900
2	2	0.35	445.5	4.5	0.15	900
3	2	0.35	441	9	0.35	900
4	2	0.35	436.5	13.5	0.50	900
5	2.5	0.35	450	0	0.20	1125
6	2.5	0.35	445.5	4.5	0.40	1125
7	2.5	0.35	441	9	0.60	1125
8	2.5	0.35	436.5	13.5	0.80	1125
9	2	0.4	450	0	0.00	900
10	2	0.4	445.5	4.5	0.15	900
11	2	0.4	441	9	0.30	900
12	2	0.4	436.5	13.5	0.35	900
13	2.5	0.4	450	0	0.10	1125
14	2.5	0.4	445.5	4.5	0.35	1125
15	2.5	0.4	441	9	0.50	1125
16	2.5	0.4	436.5	13.5	0.60	1125
17	2	0.5	450	0	0.00	900
18	2	0.5	445.5	4.5	0.10	900
19	2	0.5	441	9	0.10	900
20	2	0.5	436.5	13.5	0.15	900
21	2.5	0.5	450	0	0.10	1125
22	2.5	0.5	445.5	4.5	0.10	1125
23	2.5	0.5	441	9	0.15	1125
24	2.5	0.5	436.5	13.5	0.20	1125

3. Results and discussion

Results of compressive and flexural strength tests and also absorption test are shown in Table 3. Based on these results, we investigate the performance of silica nano-particles in the matrix of specific cementitious mortars.

According to Figures 1 to 4 which depict the compressive strength tests results for mixtures with S/CM ratios of 2 and 2.5 at 7 and 28 days, we can observe that the compressive strengths of all mixtures have increased with addition in nano-particles content. This trend has continued with increase in silica nano-particles content up to 3% replacement.

The reason for this strength enhancement pertains to fourfold behavior of silica nano-particles in the matrix of cement-based materials. As we reinforce the cement-based materials with silica nano-particles, simultaneously, four different processes occur with different intensities (depend on specific surface area of nano-particles).

These four behaviors are explained as follows: 1- nucleic behavior for developing the hydration reaction, 2- supper pozzolanic behavior, 3- micro and nano filling behavior, and 4- preventing the uncontrollable growth of defective crystals.

These four behaviors contribute to formation of a dense and strong transition zone and also enhancement of cement matrix. However, according to experimental results, this increasing trend has not been seen in some of mixtures at specific ages. We can refer this issue to various reasons.

Non-uniform distribution of nano-particles and consequent formation of unsteady clogs in the cement paste matrix, sensibility of cement-based materials reinforced with nano-particles to mix procedure and casting are some of the most remarkable reasons.

TABLE 3: RESULTS OF MECHANICAL AND DURABILITY TESTS

Mix No.	Compressive strength (MPa)		Flexural strength (MPa)	Water absorption (%)	Stability	
	7 days	28 days	28 days	28 days	7 days	28 days
1	47.9	58.7	6.5	-	3	3
2	48.5	62.7	7.1	-	4	4
3	55.5	68.2	7.9	-	4	4
4	59.3	73.3	8.9	-	4	4
5	44.1	55.7	6.4	-	3	3
6	47.9	60.5	7.0	-	4	4
7	53.4	65.8	7.7	-	4	4
8	55.6	70.5	8.4	-	5	5
9	38.7	50.8	6.1	5.02	3	4
10	42.3	54.5	6.5	4.23	4	4
11	42.5	57.1	6.8	3.92	4	4
12	51.5	62.7	7.5	3.51	5	5
13	32.2	47.3	5.8	5.12	3	4
14	37.1	48.2	6.2	4.44	4	5
15	32.3	55.2	6.6	4.12	5	5
16	47.7	59.2	6.9	3.71	5	5
17	26.4	36.6	5.1	-	3	4
18	31.1	38.9	5.4	-	4	4
19	32.1	40.3	5.5	-	4	4
20	35.2	41.8	5.7	-	5	5
21	21.9	34.4	4.7	-	3	4
22	28.3	37.4	5.1	-	4	4
23	31.1	38.5	5.3	-	5	5
24	33.5	40.8	5.5	-	5	5

As we know, nano-particles behave singularly regarding their ultra high specific surface area. This property results in both positive and negative aspects. Extremely high reactivity and quick

sensible effects of nano-particles in the matrix of materials is one positive influence, and agglomeration, accumulation, and adhesion of nano-particles to each other and attraction of water molecules are some negative effects. So, on one side, we face low strength cores in the paste matrix and also transition zone, and on the other side, a thick fresh mixture will be gained after the mix procedure.

Therefore, due to high sensibility of mixtures, in this study, we tried our best to avoid the agglomeration by using colloidal nano-particles. In addition, by utilizing low amount of nano-particles, we benefitted in both economical and operational aspects. Not to mention that the addition of water reducing superplasticizer has also been helpful in providing more homogenous mixture and better distribution of nano-particles in the matrix of cement mortars.

Yet, sensibility of mixtures to minute changes in the fabricating process has caused some unexpected results. Accordingly, we should design a mixture with high workability to prevent the formation of voids produced in the matrix of mortars during the mixing process due to thickness of fresh mixtures.

To avoid such defects, rheological properties are some of determining factors in quality control of mortar designation. The same as before-mentioned procedure, Mini Slump Flow tests were conducted to examine the similarity of different mixtures to be used as flowable ferrocement mortars. In this process, the importance of super-plasticizers is not concealed from any concrete expert regarding the financial and workability features of concrete production. As it can be seen in Table 2, by increasing the nano-particles contents, the required amount of superplasticizers increases.

Due to possessing high specific surface area and numerous unsaturated bonds, silica nano-particles are representing ultra high capability of reactivity. Accordingly, by attracting the water molecules around their surface and creating a chemical bond, they cause the formation of silanol groups (Si-OH). This matter influences the performance of free water existing in mixture which is supposed to enhance the flowability of fresh mix. As a result of this fact, the flowability of mixture faces a significant decrease [17]. Therefore, as the nano-particles replacement ratio increases, it would be more difficult for us to procure the determined flowability specifications, and higher dosages of superplasticizers are needed.

By comparing Figures 1 to 4, we can conclude that the increase in S/CM ratio results in strength loss of specimens. Formation of more surfaces of transition zone which are the weakest sections of mortars and concretes, and also reduction of binding paste enclosed by aggregates (regarding the increase in aggregate volume and constant volume of mortar) are the reasons of this fact. On the other hand, increase in W/CM ratio has weakened the promoting behavior of silica nano-particles. Because in mixtures with higher W/CM ratios, a large number of silica nano-particles locate in the aquatic environment of free water which will result in formation of voids containing agglomerated nano-particles.

According to Figures 5 and 6, resulted trend of 28-days flexural strength test is almost similar to that of compressive strength (especially for 28-days). As we are aware, all the mechanical properties of cement-based materials are partly relevant to compressive strength and the only probable dissimilarity is referred to strengthening trend of different strengths.

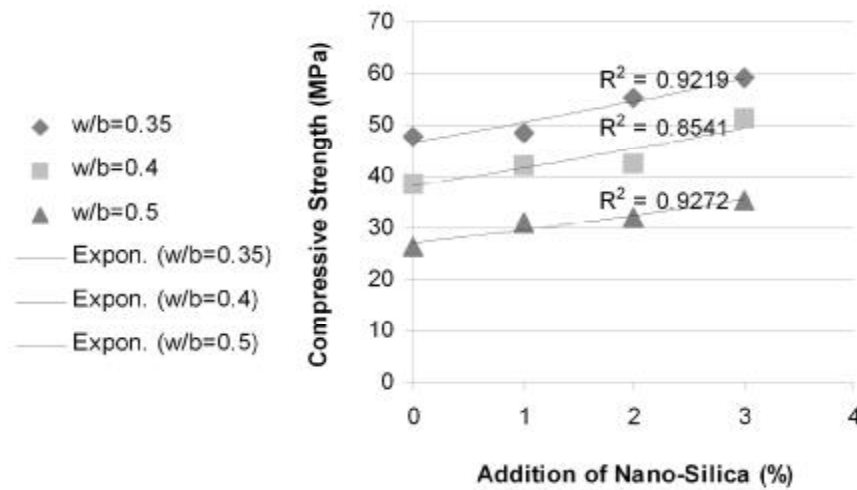


Figure 1. Compressive strength of series with sand/binder=2 at 7 days of curing

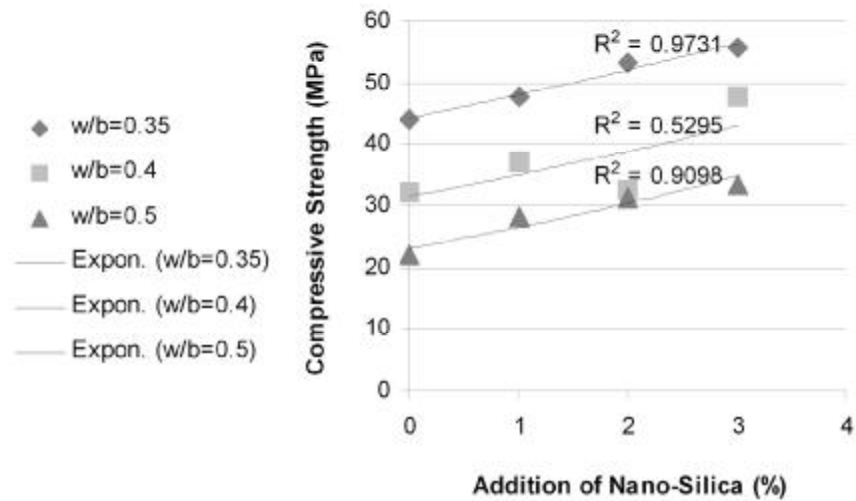


Figure 2. Compressive strength of series with sand/binder=2.5 at 7 days of curing

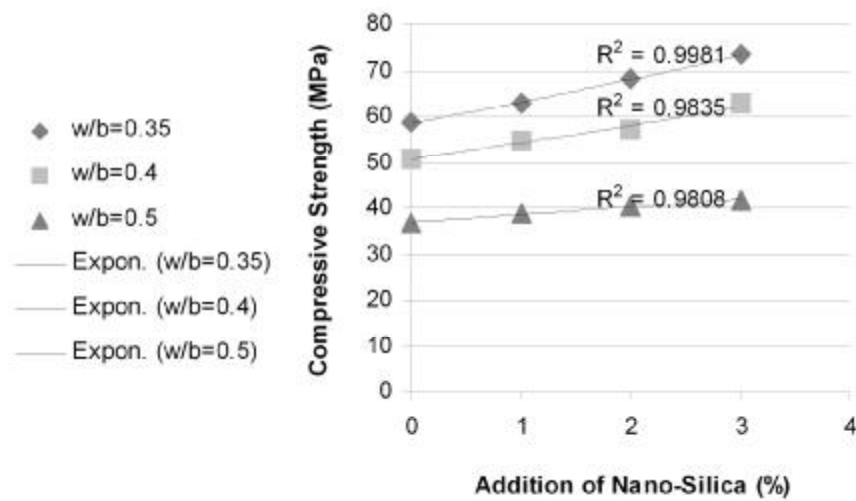


Figure 3. Compressive strength of series with sand/binder=2 at 28 days of curing

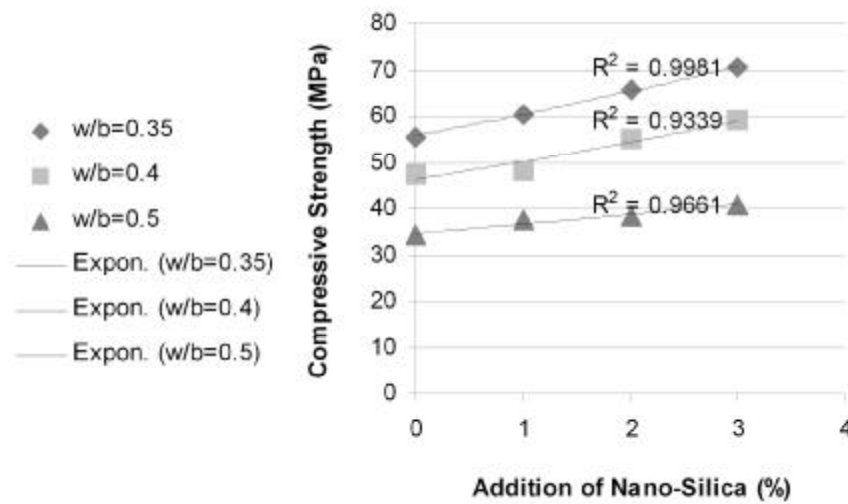


Figure 4. Compressive strength of series with sand/binder=2.5 at 28 days of curing

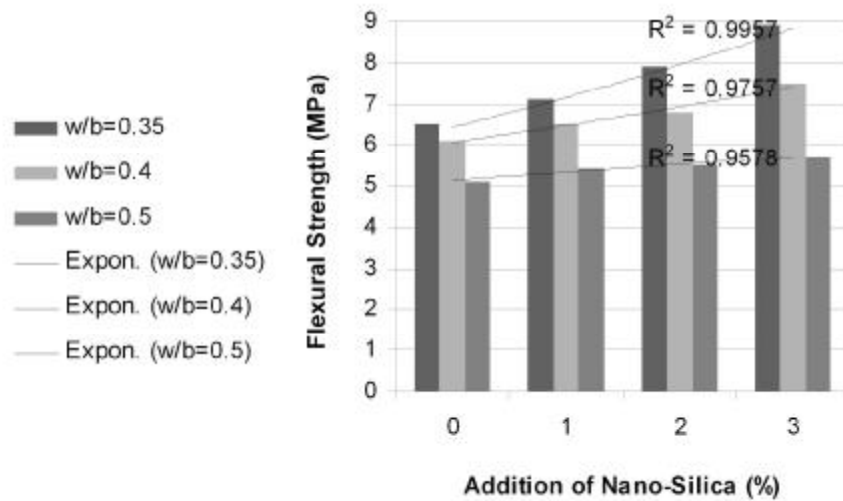


Figure 5. Flexural strength of series with sand/binder=2 at 28 days of curing

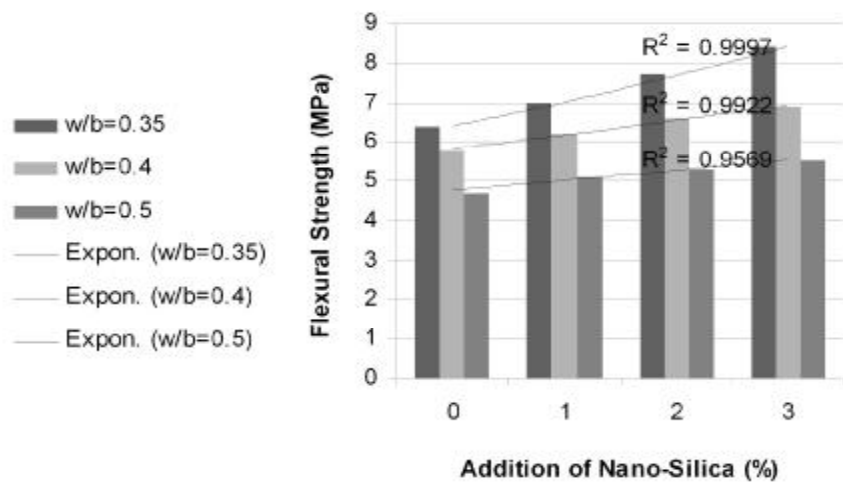


Figure 6. Flexural strength of series with sand/binder=2.5 at 28 days of curing

With a glance at the results of dimensional stability achieved by a qualitative previously-defined test, it can be observed that the stability of specimens increased with increase in silica nano-particles content (from 0 to 3%). Besides, increase in S/CM ratio has enhanced the stability. Stability improvement trend is more tangible after the compressive strength tests at 28-days.

Based on the results of water absorption test indicated in table 3, the increase in the amount of silica nano-particles will lead to decrease in water absorption of the mortar specimens. It can be a result of enhancement in permeability mechanism of mortars due to super-pozzolanic performance of silica nano-particles.

The cut in the relation between voids and their subsequent separation leads to the better permeability mechanism in the specimens. However, as can be concluded from the results, adding 3% silica nano-particles has noticeable effects on the water absorption. Furthermore, the water absorption has been heightened through the increase in proportion of sand to binder. This weakening trend is similar to that of mechanical tests. Hence, it can be expressed that the increase in the quantity of S/CM ratio does not lead to improving the resistance and durability of the specimens.

Along with the mentioned issues, and for the examination of microstructure of the transition zone of cement mortar specimens, SEM and AFM tests have been conducted on the polished specimens. Figures 7 and 8 are the SEM images of the microstructure of transition zone of mixtures 1 and 4. These figures indicate that addition of silica nano-particles has led to an improvement in the transition zone between paste and aggregates.

The ITZ structure has been improved as a result of decrease in the quantity of voids due to the micro and nano filling effect, and the decrease in the amount of Ca(OH)₂ crystals which leads to higher amount of Calcium-Silica-Hydrate gel. For more precisely examination, the AFM was used. Figures 9 and 10 are AFM results for transition zone between the paste and aggregate in the mixtures 1 and 4. According to these figures which indicate the surface morphology in scanning scale of 3*3 micrometers, usage of nano-particles has led to a smoother and denser surface in the transition zone.

This issue has double significance during the evaluation of the fractured surface; because, improvement of morphologic characteristics of a fractured surface contributes to a surface with higher fracture energy.

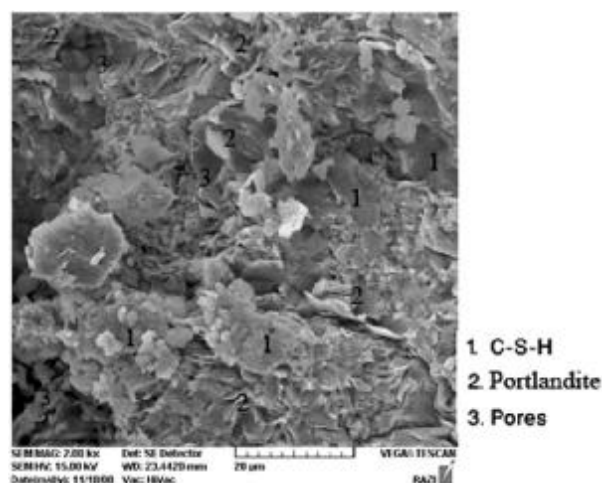


Figure 7. ITZ of Mix 1 (basic mixture without silica nano-particles)

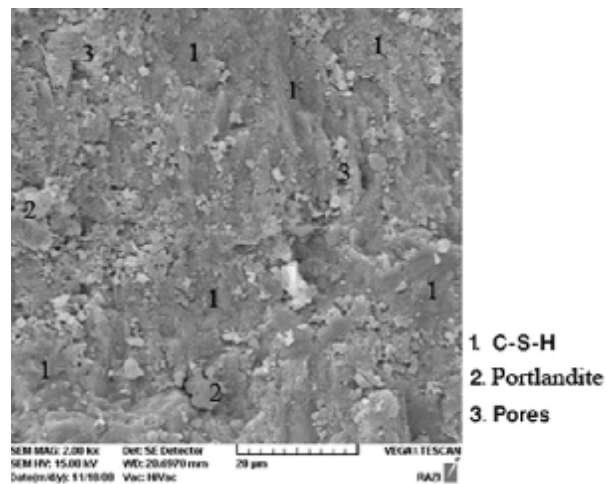


Figure 8. ITZ of Mix 4 (with 3% silica nano-particles)

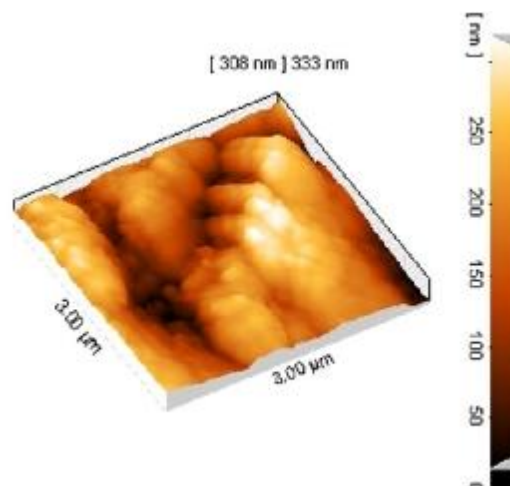


Figure 9. Morphology of fractured surface of ITZ (Mix 1)

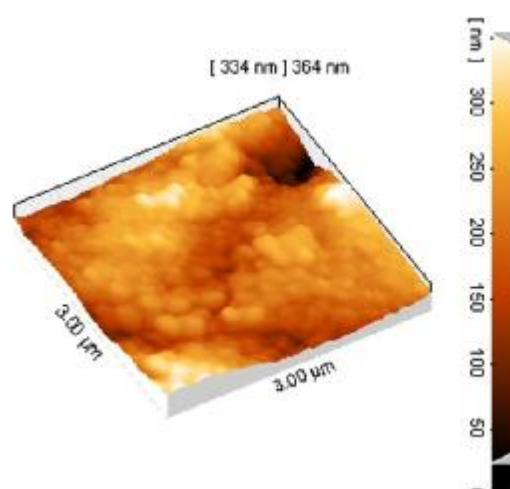


Figure 10. Morphology of fractured surface of ITZ (Mix 4)

4. Conclusion

Based on the results of this research, the below conclusions can be mentioned:

- 1- Application of silica nano-particles (in low amount of replacement up to 3%) can lead to microstructural development due to their multi functional behavior in the matrix of cement-based materials. This characteristic can enhance both durability and mechanical properties of ferrocement mortar specimens.
- 2- As the S/CM ratio lessens, the resistance and water absorption improve. In addition, increasing the W/CM ratio causes a decrease in the intensity of performance of silica nano-particles within the matrix of cement mortar.
- 3- Various performances of silica nano-particles helping resistance, durability and viscosity of cement mortars to be improved, indicate their high potential of usage in the production of special mortars such as ferrocement and retrofitting mortars.
- 4- Microstructural development of transition zone and fractured surface of mortar specimens is the result of fourfold behavior of silica nano-particles into the matrix of cement mortar that are prevention of growth of harmful crystals such as Ca(OH)₂ and AFm, more production of C-S-H gel, micro and nano filling effect, and helping the hydration reaction to be developed due to nucleus-like action.

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